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Title: Pulsed Neutron Scattering Technique and Uncertainties

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Pulsed Neutron Scattering Technique and Uncertainties

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Disclaimers & Full Disclosure

A. I am employed by Los Alamos National Laboratory-

<u>Material Science Division: Extreme Scattering Team</u>

   A. I support pulsed neutron material science beamlines
   B. I develop and deploy novel in-situ sample conditions
   C. I develop in-situ techniques at national light sources

B. I seek to better understand the limitations of the instruments

C. I want to raise awareness of my work to others

I work here

Agenda

- Motivation and Background:
  - What is the “Pulsed Neutron Scattering Technique”
  - Relevance to Physical Metallurgy, Why we care, and why it’s used
- Uncertainty Analysis: Peak analysis
  - Data and Peak fitting
  - Stochastic <u>uncertainty</u> vs Systematic <u>error</u> sources

How do we Actually take non-destructive measures of d-spacing of bulk materials?

-What is our “confidence” in our measurement

Note: Neutrons are the ONLY source for true bulk measurements

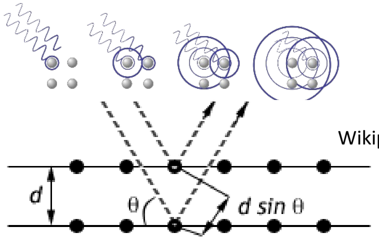
Photon penetration depth: ~$Z^{1/3}$ {Of order mm for Actinide elements

Electron probe depth: 275 μm

Proton scattering not used because it transmutates the nucleus

First: What are we talking about-> Bragg's Law

$$\underbrace{n\lambda}_{\text{Source}} = \underbrace{2d}_{\text{Sample}} \underbrace{\sin\theta}_{\text{Detector}}$$



Wikipedia image

Second: With what? -> Neutrons

-Use the dual nature of matter: waves AND particles

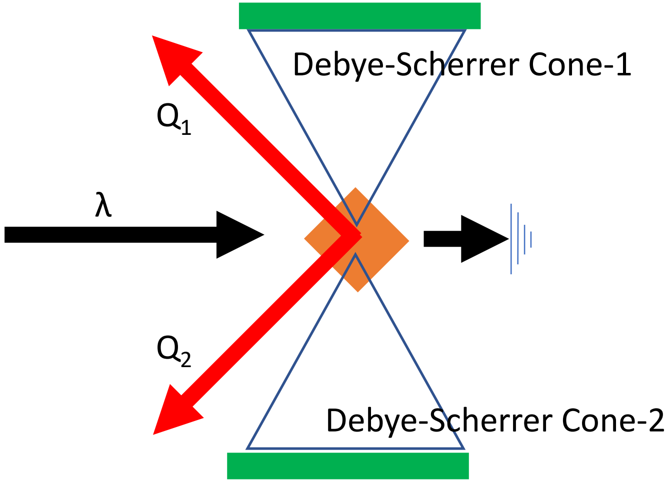
$$\lambda = \frac{E}{c} = \frac{h}{\gamma m_0 v} = \frac{h}{m_0 v}\sqrt{1 - \frac{v^2}{c^2}}$$

But how do you really do it?

-Samples are polycrystalline, bulk measurements

-Fix scattering vector-Q_1&Q_2

-Scan over Wavelength



But you said Pulsed Neutrons…

-Neutrons are produced from a particle physics reaction:

$$ {}^{1}_{1}p^{+} + {}^{184}_{74}W = {}^{*}A + {}^{*}B + {}^{0}_{0}\gamma + \sum {}^{1}_{0}n\ (E_i) $$

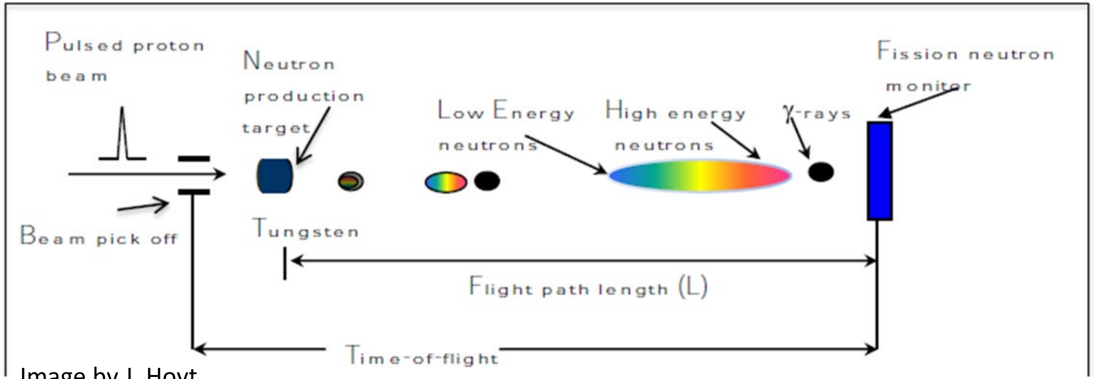
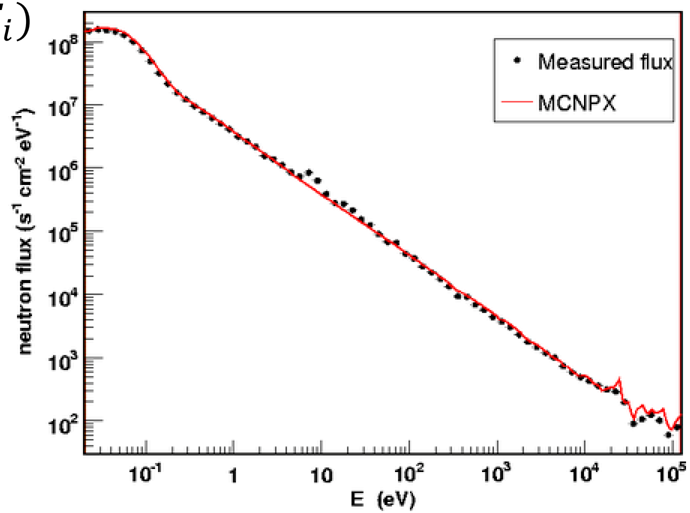


Image by J. Hoyt



Each Proton striking Tungsten spalls ~25 neutrons that emit over a range of Energies over 4*Pi (sphere)
-Because these particles have Mass they travel at speeds LESS than light (borderline relativistic)
-Thus High energy neutrons travel faster than lower energy neutrons
-Wavelength is directly proportional to Energy

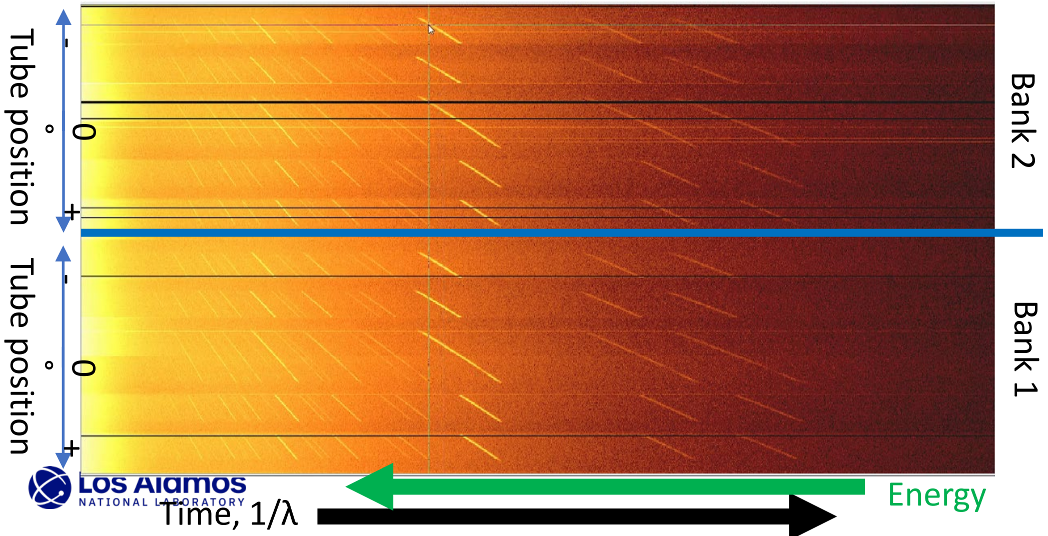
Why do we care? This isn't instrument Physics design

Because we study engineering material, polycrystalline, how does d-spacing change due to:

- Coefficient of Thermal Expansion
- Activated Slip systems under "Conditions"
- Mobility & diffusion mechanisms

(Yes, I'm glossing over things)

- **Residual Stress**: The unrealized deformation of the metal $\varepsilon_i = \frac{d_i - d_0}{d_0}$ measured as strain
  - Stress from Hook's law $\sigma_i = \frac{E}{(1+v)(1-2v)}\left((1-v)\varepsilon_i + v(\varepsilon_j + \varepsilon_k)\right)$

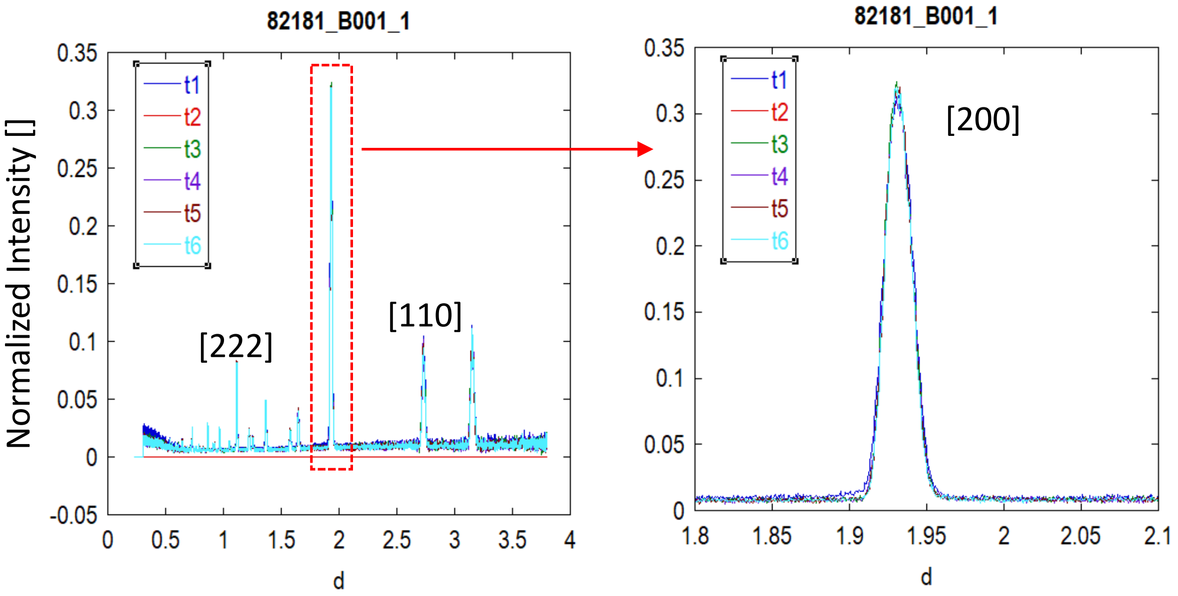


Raw Data displayed from "Area" Detectors
-Banks 1 & 2
-"Area" Detectors are actually 192 He-3 tube

We are looking at the Debeye-Scherrer Cones over TIME (that equal wavelength) as it continually satisfies the Brag diffraction for those Energies/wavelengths

How do we quantify and apply uncertainty

- CaF_2 is our d-spacing calibration sample
  - Cubic: a=3.9Å space group $Fm\bar{3}m$
- We now assign and calibrate ***Each Tube*** to the known d-spacing



The canister also emits peaks

Now we can start to estimate uncertainty

Peaks give us additional information

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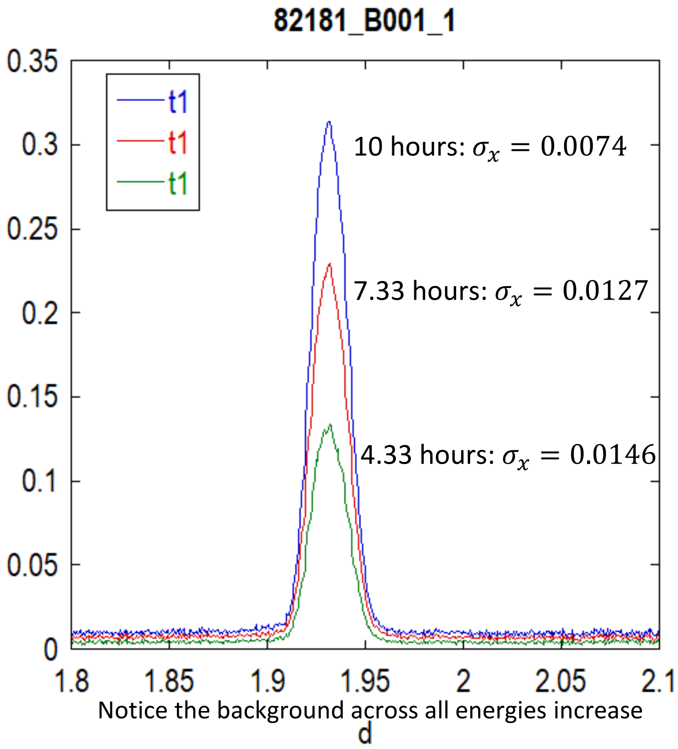
Perfect, “exact” data would give us Dirac-delta function peaks, we have additional ‘features’

Appling uncertainty statistics

- Lets start by assuming a normalized Gaussian Distribution*
    - $f(x) = \frac{1}{\sigma\sqrt{2\pi}} exp\left[-\frac{(x-x_0)^2}{2\sigma^2}\right]$
- Measure for different lengths of time
- Calculate FWHM [Full Width at Half Maximum]
    - $FWHM = 2\sqrt{2ln2}\sigma_x$
- Calculate Standard Error [estimate]
    - $\sigma_{\bar{x}} = \frac{\sigma}{\sqrt{n}} \approx \frac{\sigma_x}{\sqrt{n}}$

For well behaved peaks (or intense) this shows expected intensity growth and FWHM growth

Where does this come from?

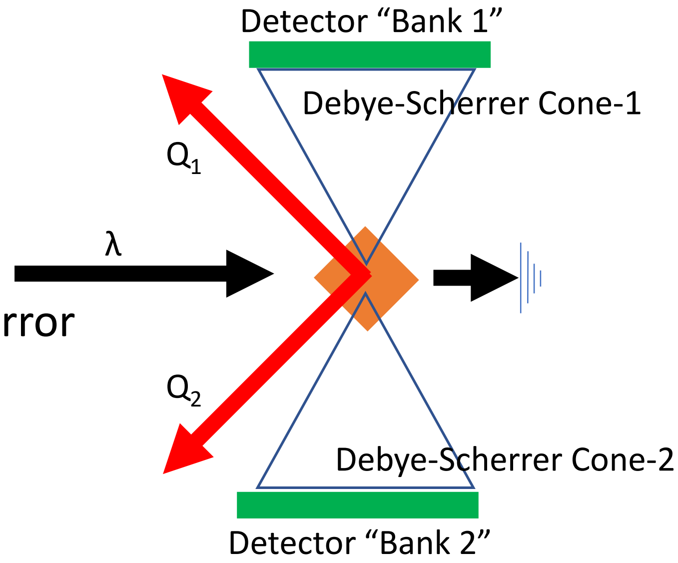


Notice the background across all energies increase

*Peaks not strictly Gaussian: used to facilitate the approximation of error

Finally: Where does the Uncertainty and Error come from?

$$\underbrace{n\lambda}_{\text{Source}} = \underbrace{2d}_{\text{Sample}}\ \underbrace{\sin\theta}_{\text{Detector}}$$



Peak Width is NOT strictly uncertainty and error

1) Error: Sample alignment
2) Error: Detector Bank
3) Uncertainty: Neutron Energy
4) Uncertainty: Inelastic Scattering
5) Uncertainty: Secondary Sample Scattering
6) Uncertainty: Air scatter

Conclusions and acknowledgments

- Neutron scattering (diffraction) is still a relevant choice in Metallurgy toolbox
- Uncertainties still less than most stated lattice parameters
- Unrealized strain (and therefore stress) can be mapped over the ‘part’ geometry

- Assisted by Bjorn Clausen, Sven Vogel, Alexander Long

References

- Roger Pynn: “Neutron Production & Scattering Primer”
- G. L. Squires: “Introduction to the theory of Thermal Neutron Scattering”
- T. E. Mason et al., "The Spallation Neutron Source: A Powerful Tool for Materials Research,"